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BENEFICIATION OF MAGDALEN SAND FOR FOUNDRY, GLASSMAKING AND BUILDING MATERIAL UTILIZATION

G. I. MATHIEU

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BENEFICIATION OF MAGDALEN SAND FOR FOUNDRY, GLASSMAKING AND BUILDING MATERIAL UTILIZATION

by

G.I. Mathieu*

FOREWORD

Canada relies heavily on imported silica sand for its requirements. About half of eastern Canada's annual silica consumption, i.e., about 1.2 Mt, is imported from the United States. Since there is only one sand supplier in the east, such a dependance on imported silica sand could become critical if the supply were to decrease. Although of good quality, the present Canadian sand reportedly carries too high a proportion of fine particles which can produce undesirable effects on the glass furnace structure and could be hazardous for the health of the workers, if sufficient caution were not exercised (1). The situation regarding the supply of foundry sand is even more critical, there being no large producer in eastern Canada. Research for exploiting domestic silica sources is therefore imperative. This present study was undertaken for Magdalen Silica Sand Inc. on a cost recovery basis.

* Research Scientist, Mineral Processing Laboratory, Canada Centre for Mineral and Energy Technology, Energy, Mines and Resources Canada, Ottawa, K1A 0G1.

ABSTRACT

Important sand deposits located in shallow water northeast and southwest of the Magdalen Islands, were recently identified and claimed by Magdalen Silica Sand Inc. The proven reserves are said to be 100 Mt, with additional sand being transported every year from the north by ocean currents. The Magdalen samples investigated contained 88% quartz, 10% feldspar, with the remaining 2% being composed mainly of sea shells, heavy minerals, and limonite coating on the quartz and feldspar grains.

CANMET'S efforts for the economic beneficiation of these marine sands have been very successful. A process was developed on a laboratory scale and demonstrated on a pilot-plant scale for recovering, at minimal cost, several commercial silica and feldspar products. The treatment avenue involved froth flotation and sizing for the production of steel foundry sand, followed by acid leaching for glassmaking grades. Other products, obtained at various stages, would be suitable for sand blast applications, as abrasives, and as extenders in certain products.

The novel approach to the beneficiation of silica sand is economically feasible in that it:

- eliminates the use of conventional, costly and hazardous hydrofluoric acid in flotation;
- utilizes relatively inexpensive sulphuric acid both in the flotation and the leaching steps;
- uses moderate temperature and rather short retention time during acid leaching along with multiple recirculation of the liquor.

VALORISATION DU SABLE DES ÎLES DE LA MADELEINE POUR UTILISATION COMME MATÉRIAU DE FONDERIE, DE FABRICATION DU VERRE ET DE CONSTRUCTION

par

G.I Mathieu*

AVANT-PROPOS

Le Canada compte fortement sur les importations de silice pour combler ses besoins. Environ la moitié de la silice utilisée annuellement dans l'est du Canada, i.e., à peu près 1,2 tonne métrique est importée des États-Unis. Comme il n'y a qu'un fournisseur de sable dans l'est, une telle dépendance sur les importations de sable quartzeux pourrait s'avérer dangereuse si les approvisionnements en silice diminuaient. Bien qu'il soit de bonne qualité, le sable canadien exploité actuellement possède une proportion trop élevée de particules fines qui ont des effets indésirables sur la structure vitreuse du four et qui posent des risques pour la santé des travailleurs si des précautions suffisantes ne sont pas prises (1). La situation des approvisionnements en sable de fonderie est même plus sérieuse puisqu'il n'y a aucun fournisseur important dans l'est du Canada. Par conséquent, il est urgent que des projets de recherche portant sur l'exploitation de la silice au pays soient élaborés. Le rapport présente une étude entreprise par le Magdalen Silica Sand Inc. sur une base de récupération des coûts.

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*Chercheur scientifique, Laboratoire du traitement des minéraux, Centre canadien de la technologie des minéraux et de l'énergie, Énergie, Mines et Ressources, Ottawa, K1A 0G1

RÉSUMÉ

D'importants dépôts de sable situés en eau peu profonde au nord-est et au sud-ouest des Îles de la Madeleine ont récemment été identifiés et revendiqués par la Magdalen Silica Sand Inc. On évalue les réserves prouvées à 100 tonnes métriques et des dépôts additionnels de sable sont apportés du Nord chaque année par les courants marins. Une investigation des échantillons du sable des Îles a révélé que celui-ci contenait 88 % de quartz, 10 % de feldspath et, pour le reste, environ 2 % de coquillages et de minéraux lourds et qu'une couche de limonite recouvrait les grains de quartz et de feldspath.

Les travaux de CANMET portant sur la valorisation économique des dépôts marins des Îles ont obtenu beaucoup de succès. Un procédé de récupération peu coûteux de plusieurs produits commerciaux dérivés de la silice et du feldspath a été mis au point à l'échelle du laboratoire et une démonstration en a été faite en usine expérimentale. Le traitement comprenait la flottation par mousse et le classement volumétrique pour la production de sable de fonderie suivi de la lixiviation à l'acide pour l'obtention d'un sable propre à la fabrication du verre. Certains autres produits obtenus à diverses étapes du traitement pourraient être utilisés pour le nettoyage au jet de sable, comme abrasifs ou comme produits complémentaires.

Le nouveau procédé de valorisation des sables quartzeux est économiquement rentable pour les raisons suivantes:

- il élimine l'utilisation de l'acide fluorhydrique du procédé classique de flottation et par le fait même, les coûts élevés et les risques qui en découlent;
- il requiert l'utilisation de l'acide sulphurique relativement peu coûteux dans les procédés de flottation et de lixiviation;
- au cours de la lixiviation à l'acide, il ne requiert qu'une température moyenne, a une période de retention plutôt courte et permet la recirculation multiple de la solution.

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SUMMARY OF PROCESSES AND RESULTS

Several approaches for treating the Magdalen sand were initially tested on a laboratory scale, including scrubbing, multiple flotation, magnetic separation and acid leaching. The flowsheet adopted integrated two flotation circuits, i.e., one for the recovery of foundry sand at minimal cost by the removal of feldspar using starvation amounts of a single collector; the other for the production of glass sand by a new combination of anionic-cationic reagents to float simultaneously both feldspar and some iron-bearing minerals. Further upgrading of the silica to glass grade requirements was achieved essentially by acid leaching and/or magnetic separation. A summary of the degree of processing with some corresponding analyses of the products, is shown in Table 1.

TABLE 1: Analysis of raw and processed sands

Products		Processed Silica Sand			Feldspar Flotation Concentrate (5)
Analysis	Washed Raw Sand (1)	Flotation (2)	+H_2SO_4 Leach (3)	+HCl Leach or Mag Sep (4)	
SiO_2	96.1	99.3-99.5	99.6	99.7	68-72
Al_2O_3	1.9	0.25-0.20	0.20	0.15	15-16
Fe_2O_3	0.2	0.12-0.09	0.03	0.01-0.02	0.7-0.6
Heavy Minerals and Middlings	0.05-0.10	0.02-0.04	0.01-0.02	<0.01	≈0.5

Product (1) is satisfactory for iron foundry sand and glass fibre production; product (2) is suitable for the production of coloured glass containers and for steel foundry sand (it was tested with success in the latter application); product (3) is suitable for the production of colourless and clear flat glass; product (4) meets the requirements of some special glassware and approaches optical grade requirements (0.01 to 0.008% Fe_2O_3). Finally, the feldspar flotation concentrate is suitable for building materials and can be further upgraded to glass and ceramic grade.

INTRODUCTION

CANADIAN IMPORTS OF SILICA SAND

Demand for quality quartz sand is expected to increase due to new applications in telecommunication, solar energy cells, chemicals, and a return to glass containers and glass fibre insulation. Canada traditionally imports large quantities of silica sand, mainly from Illinois and Michigan, USA. The price of imported sand is steadily increasing due mainly to higher transportation costs. Further to higher demand and price for silica, a shortage of railway cars may develop in the USA due to the high priority given to coal transportation. It is imperative therefore that Canada becomes more self sufficient with regard to domestic silica requirements.

TABLE 2: Canadian imports of silica sand*

Year	Mt	M$	$/T
1977	1.10	9.50	8.64
1978	1.24	13.50	10.89
1979	1.65	19.40	11.74
1980	1.18	17.37	14.67
1981	1.23	18.51	15.10

* After Statistics Canada (FOB Canadian border)

With the additional Canadian freight cost, the silica sand price already attained $40.67/t in Toronto and $41.88/t in Montreal in 1979 (2).

BACKGROUND INFORMATION

Origin and Nature of Sand

Sands consists of grains of 0.1 to 1 mm size which have been produced by the weathering and breakdown of rock. They mainly are composed

of quartz, feldspar and mica minerals, with minor amounts of zircon, garnet, glauconite, tourmaline, kyanite, rutile, ilmenite, magnetite, hematite and limonite. The limonite appears as thin coating on the silicate surfaces. Quartz (SiO_2) is the principal component of most sands although some may essentially consist of minerals other than quartz, e.g., olivine and zircon.

Definition and Uses of Silica Sand

"Silica sand is a term used by industry to describe a material which contains a high degree of silica in the form of quartz and which is marketed principally for glassmaking and foundry purposes (3)." Quartz sands of a purity suitable for glass and foundry sand applications do not normally occur in nature. Most sands must be beneficiated to satisfy these requirements. Other important applications of silica include optical glass, solar cells, fiberoptics, and in the manufacture of chemicals and insulation products. Chemical composition is the main consideration for the glassmaking and chemical manufacturing industries whereas, in the foundry and building material applications, physical characteristics are important.

Specifications for Silica Sand

The value of a foundry sand is determined by its minerology and mechanical grading, i.e., its composition and the shape and size of its grains. The mineralogical composition determines the degree of refractoriness, an important factor in casting molten metals. For instance, a steel foundry sand must contain 97 to 99% SiO_2 of which at least 95% must be in the form of quartz and the remainder as hard silicates. Both the clay and alkali silicates must be below 1% for steel casting. On the other hand, a sand with a 90 to 95% quartz content is satisfactory for iron and some non-ferrous casting. To determine whether a sand has adequate refractoriness for casting, its "B" sintering point is determined by a conventional method indicating at which temperature the sand "burns on" or adheres to a hot casting surface. Minimum sintering temperatures, for example, are 1260°C for non-ferrous metals, 1316 to 1454°C for gray, ductile and malleable iron, and 1427 to 1538°C for steel castings (4).

An ideal foundry sand according to Davies should be composed entirely of round grains for a maximum permeability of the gases formed

during the casting (5). On the other hand, Sanders considers that a certain angularity is advantageous for maximizing the strength in bake cores, albeit more bond and other additives are required (6). Sands with an angularity coefficient of up to 1.20 are now accepted by foundries.

A well-graded sand has 90% of its grains within three successive US sieve fractions with more than 80% between 200 and 400 µm. Although it is realised that it is standard practice both in foundry and glassmaking to use the US sieve series, all sizes quoted in this report are metric. To assist in comparing with metric sizes, commonly used US sieve sizes are shown with the corresponding metric sizes in Table 3. This table also reports typical screen tests on the Magdalen sand head sample which illustrate its satisfactory mechanical grading.

TABLE 3: Comparison of US and metric sieve series with screen sizes on Magdalen raw sand

Tyler Mesh	28	35	50	65	100	150	200
U.S. Standard (No.)	30	40	50	70	100	140	200
Metric System:							
Number	590	420	297	210	149	105	74
Opening (µm)	590	420	297	210	149	105	74
Magdelen Screen Tests:							
% retained (CANMET)	0.1	3.0	33.8	47.6	14.5	0.5	0.0
% retained (Dietert*)	0.0	1.0	34.8	53.1	10.2	0.2	0.1

* H.W. Dietert Co., "Test Report No. T-6114", Detroit, Michigan, January 28, 1982.

The most important system currently used for classifying foundry sand is the "AFS Grain Fineness Number". It is determined as follows:

a) take a representative sample and wash the clay minerals;

b) ro-tap on a standard series of US sieves;

c) take the weight retained on each screen and multiply it by the screen number just passed by the grains;

d) sum all the figures so obtained and divide by the total grain weight.

The quotient is an average size figure termed the AFS Grain Fineness Number (7). The calculation method is illustrated in Table 4 using screen data from the Magdalen raw sand.

TABLE 4: AFS grain fineness number of Magdalen raw sand

U.S. Series Equivalent Number	Amount of 100-g Sample Retained on each Sieve	Multiplier	Product
30	0.1	20	0.2
40	3.0	30	90.0
50	33.8	40	1352.0
70	47.6	50	2380.0
100	14.5	70	1015.0
140	0.5	100	50.0
200	0.0	140	0.0
AFS Clay	0.6	-	-
TOTAL	100.0	-	4837.2

$$\text{AFS Fineness Number} = \frac{\text{Total Product}}{99.4} = 48.7$$

Sands with an AFS number of 50 and upward are qualified as "fine", whereas those below 50 are termed "coarse" (8). The Magdalen raw sand is thus slightly coarse. It can be made to match standard Ottawa sand by removal of some of its top size.

A summary of the most desirable properties of foundry sands is given in Table 5.

TABLE 5: Summary of foundry sands requirements

Physical Properties	Criteria
Suitable Mineralogical Composition	Steel: 97-99% SiO_2 (total) 95% quartz; less than 1% clay and alkali feldspars. Iron and non-ferrous: 95-96% SiO_2 (total); 90-95% quartz; K, Na feldspars low enough to stay over sintering temperature
High Sintering Temperature	+1260°C for non-ferrous 1316-1454°C for gray and malleable iron 1427-1538°C for steel
Acceptable Grain Fineness	AFS No. 45 to 55
Spherical or Semi-angular and Well Graded	Angularity coefficient less than 1.20 and size range within 420 µm to 105 µm.

In contrast to foundry applications which rely mostly on the physical characteristics of sand, the glass industry is mainly concerned with the chemical composition of the sand and by the level of some impurities. A silica sand used in the clear glassmaking industry shall contain a minimum of 98% quartz with 99% of the grains smaller than 600 µm and 95% coarser than 120 µm (9). The main impurities in glass sands that are particularly critical to glass manufacturing are the heavy oxides of iron, titanium, and chromium which impart unwanted colour to the glass. These undesirable contaminants might occur either as coatings on the quartz grains or as discrete particles. The ferruginous minerals, and particularly the chromite, are much less harmful if present as staining on the quartz rather than as liberated particles. The latter might not fuse in the melt and may cause black specks or "stones" in the glass. The iron, chromium and other impurity specifications for glassmaking sands are noted in Table 6.

TABLE 6: Chemical composition requirements of glassmaking sands*

Constituent Requirements	Type of Glass Product			
	Insulation Fibres	Coloured Containers	Clear Flat	Colourless Containers
Silica: minimum % SiO_2	94.5	97.0	99.0	98.8
Alumina: maximum % Al_2O_3	3.0	Nominal**	0.5	Nominal**
Total Iron as Ferric Oxide: maximum % Fe_2O_3	0.3	0.25	0.10	0.03
Total Chromium as Chromic Oxide: maximum % Cr_2O_3	-	-	-	0.0005
Loss on Ignition: maximum wt %	0.5	0.5	0.2	0.2

*Chemical Analysis Committee, "Specifications for and recommended procedures for the testing and analysis of glassmaking sands 1978", Glass Technology, Vol. 19, No. 5, October 1978:93-101.
**Fixed by agreement (usually 0.2 to 0.3% Al_2O_3).

MAGDALEN SAND STUDY

PURPOSE OF INVESTIGATION

The specific goals of this study were: 1) the production of satisfactory steel foundry sand, and 2) the purification of the silica sand to meet both coloured and clear flint glass specifications. A cursory examination of the Magdalen raw sand sample indicated that the first objective could be met by removing the alkali silicates from the raw sand, whereas the second goal would also require the removal of harmful metal oxides and iron coating on the quartz grains. CANMET's investigation was thus undertaken to achieve these aims.

ANALYTICAL METHODS

In this work, the determination of low contents of alumina, iron oxide, titanium oxide, etc., in the silica products was made by optical

emission spectrometry. The higher alumina and iron values, such as in the flotation concentrates, were either calculated by difference from the head sample or determined by X-ray analysis. Finally, the intermediate levels, e.g., head sample, sized fractions and cleaner flotation tailings, were determined by wet chemical analyses. This last technique also was used periodically to verify the instrumental determinations.

CHEMICAL AND MINERALOGICAL ANALYSIS OF RAW SAND

A.E. Gregory, a specialist in sand processing, wrote: "before any attempt can be made to design a processing plant for a sand, a detailed chemical and mineralogical study should be made... If the distribution of the impurities in the sand is known, it is possible to predict which method of treatment is likely to yield the best results. In this respect, every sand must be considered on its own (10)."

A representative head sample was cut from several bags of a 2 t shipment received from Magdalen Silica Inc. of Montreal, Quebec. A portion was pulverized in an agate mortar and chemically analysed. The analytical results are shown in Table 7.

A 500-g sample was riffled from the head sample and screened using a conventional US Standard series of sieves. The sized fractions were weighed and analysed for alumina and iron oxide. The results are shown in Table 8.

A number of dark particles were observed in the fine fraction, i.e. -149 µm. Its titanium and chromium oxide contents were therefore determined. These were 0.6% and 55 ppm, respectively, thus confirming the high level of undesirable impurities in the fines.

To obtain maximum mineralogical information on the Magdalen sand, a 500-g sample was first separated into three sized fractions, namely, -590+297 µm; -297+180 µm; and -180 µm. Each fraction was subsequently subjected to sink-float analysis at sp gr 2.7. The respective sinks and floats were then treated by high intensity magnetic separation. It was observed that most of the sinks were magnetic, however, the floats fractions also contained a significant amount of magnetics. The weight distributions of the sized non-magnetic float, magnetic float, and sink fractions are reported in Table 9.

TABLE 7: Chemical analysis of Magdalen head sample

Analysis	Symbol	%
Silica:		
total	SiO_2	95.0
quartz	SiO_2	approx. 88.0
Silicates:		
aluminum	Al_2O_3	2.17
potassium	K_2O	1.13
sodium	Na_2O	0.30
calcium	CaO	0.10
(all as oxides)		
Metal Oxides:		
iron (as ferric oxide)	Fe_2O_3	0.23
titanium (as oxide)	TiO_2	0.06
magnesium (as oxide)	MgO	0.15
chromium (as chromic oxide)	Cr_2O_3	9 ppm
Lost On Ignition	LOI	0.40

TABLE 8: Screen test of Magdalen head sample with size-distribution of Al_2O_3 and Fe_2O_3

Size in Microns	Weight (%)	Analysis (%)		Distribution (%)	
		Al_2O_3	Fe_2O_3	Al_2O_3	Fe_2O_3
+590	0.2*	-	-	-	-
-590+297	33.0	1.67	0.18	24.9	25.4
-297+210	47.5	2.32	0.20	50.0	41.2
-210+149	18.3	2.84	0.33	23.4	25.9
-149	1.0	3.72	1.72	1.7	7.5
TOTAL	100.0	2.21	0.23	100.0	100.0

* mostly sea shell

TABLE 9: Weight distribution of Magdalen sand by size, gravity, and magnetic properties

Fraction		Size (microns) -590+297	-297+180	-180	Total
Non-magnetic Floats (less than 2.7 sp gr)		29.2	57.7	4.9	91.8
Magnetic Floats	Sala	2.4	3.4	0.6	6.4
	Frantz	0.6	0.8	0.2	1.6
	Total	3.0	4.2	0.8	8.0
Sinks (2.7 sp gr)		0.07	0.07	0.07	0.21
TOTAL		32.27	61.97	5.77	100.00

The above four products were submitted to the Mineralogy Section for identificaton of the main components and the establishment of the component liberation characteristics. Results are summarized in Table 10.

The non-magnetic float fraction analysed 0.10 Fe_2O_3 (mostly as oxide coating) thus indicating the necessity of acid leaching for the production of the clear glass grades.

LABORATORY INVESTIGATION

The approach used in developing a flowsheet for treating the Magdalen sand consisted of minimizing the number and cost of the unit operations in order to render the whole process as economically attractive as possible. The conventional methods which use double flotation techniques (one for feldspar and one for iron minerals) both preceeded by alkali

scrubbing were not used but rather a novel flotation technique which employs one promotor for the removal of both feldspar and several iron minerals. This was sufficient for meeting the foundry grade, but the use of a second collector for further removal of metal oxides appears preferable for the glass sand. These reagents do not necessitate the use of a silica depressant (normally HF) provided the pH is rigidly controlled within a narrow range during conditioning.

TABLE 10: Mineralogical composition of Magdalen sand (11)

Product	Weight (%)	Mineral	Estimated Content (%)
Light Fractions: i) Non-Magnetic	91.8	Quartz	80
		K-feldspar	6
		Na-feldspar	2
		Quartz-feldspar (intergrowth)	2-4
		Ca-feldspar	0.2
		Clay-limonite coating (on feldspars and quartz)	0.2
		Ilmenite, chlorite, rutile (as fine inclusions in silica and silicates)	0.1
ii) Slightly Magnetic (Sala conc)	6.4	Entrained quartz	2.0
		Quartz and feldspars (with iron-bearing inclusions)	2.0
		Altered feldspars	2.0
		Iron-bearing silicates	0.4
iii) Moderately Magnetic (Frantz conc)	1.6	Fe - rich clay mineral	0.1
		Fe, Mg, K, Al, Si biotite	0.1
		Fe, Mg, Al, Si pyroxene	0.1
		Quartz and feldspars (intergrown with iron minerals)	1.3
Heavies: Highly Magnetic (except rutile)	0.2	Ilmenite	< 0.15
		Chlorite	< 0.05
		Rutile	< 0.05
		Silicate-iron oxide middlings	< 0.05
		Chromite (spinel)	Tr

Flotation Tests

Prior to every flotation test, the -590 μm and +149 μm fractions were discarded since this material was high in impurities and accounted for only 1.2% of the sand by weight. This operation also permitted the removal of salt water from the sample.

HF versus non-HF flotation method

The initial series of tests was designed to compare the new flotation approach with the conventional HF method. The conditions and reagents used for one typical test with each flotation technique are given in Table 11 and the corresponding results compared. Both tests were made on a 1000-g sample using a 500-g Denver glass cell with the impeller set at 1200 rpm for conditioning and 1600 rpm for flotation. Limited air was used in the latter to prevent sanding of the cell.

TABLE 11: HF versus non-HF flotation

I - Reagents and conditions

Operation	Time min	Reagents kg/tonne H_2SO_4	HF	TAA	PS	Density % solid	pH
HF Method:							
Conditioning	5	1.0	1.0	1.0	1.0	65	2.2-2.3
Flotation	4½					33	2.6-2.8
Non-HF Method:				PDD			
Conditioning	5	1.5	-	0.5	-	65	2.0-2.2
Flotation	3					33	2.4-2.5

II - Results obtained

Test	Product	Weight (%)	Analysis % Al_2O_3	Fe_2O_3	Distribution % Al_2O_3	Fe_2O_3
1 (HF)	Al-Fe conc*	16.5	10.4	0.75	90.0	65.2
	SiO_2 conc**	83.5	0.23	0.08	10.0	34.8
	Feed (c)	100.0	1.91	0.19	100.0	100.0
2 (Non-HF)	Al-Fe conc	16.8	10.8	0.75	90.6	61.4
	SiO_2 conc	83.2	0.23	0.10	9.4	39.6
	Feed (c)	100.0	2.01	0.21	100.0	100.0

* Froth
**Sink

TAA = tallow amine acetate
PDD = N-tallow-1,3 propanediamine dioleate (neutralized with NaOH)
PS = high molecular weight petroleum sulphonate

These results show that the non-HF method could be as effective as the classical approach while using less hazardous chemicals. The lower consumption of collector is also important. It was thus decided to pursue the investigation solely with the non-HF approach.

Important variables in non-HF flotation

During the years 1976 and 1979, Dr. S.E. Malghan studied the basics of a reagent system for the flotation of feldspar from quartz in the absence of hydrofluoric acid (12,13). Among his findings, the following were of greatest importance:

a) A different feldspar collector was necessary and the optimum level must be established.
b) A high density conditioning step of about 5 min was required prior to flotation.
c) Both conditioning and flotation had to be carried out between 1.7 and 2.6 pH.
d) The non-HF method was more satisfactory for particles less than 420 µm.

It was found that a commercial propanediamine dioleate was a satisfactory feldspar-iron collector in the absence of fluorine ions. The collector concentration was tested at various levels under appropriate condition of pH. The control of the latter gave some difficulties since the Magdalen sand contained some acid consuming minerals. This problem was resolved by adding a slight excess of sulphuric acid during the conditioning stage. The resulting low pH was not of a duration sufficient to hinder flotation, as shown in Table 12.

These results indicate that an increase of sulphuric acid improves the selectivity of the quartz-feldspar separation (see Al_2O_3 analyses in flotation concentrates), while a higher collector concentration increases the recovery of both feldspar and iron albeit with some additional loss of silica.

Optimal conditions would thus consist of a compromise between the acid addition and the level of collector. There was some difficulty in finding this optimum since, according to P. Neal (14), the two factors are interrelated. Furthermore, the addition of fresh make-up water for diluting the pulp prior to flotation rendered the results somewhat erratic from test to test.

A series of tests was then made in which the make-up water was acidified to the same pH as the end point of the conditioning step. Tests were made in the vicinity of pH 2.3 to 2.4 which is theoretically considered as the optimum for the separation of feldspar from quartz. The conditions of the tests and the results obtained are shown in Table 13.

TABLE 12: Effect of reagent concentration on feldspar-iron flotation

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne H_2SO_4	Reagents-kg/tonne PDD	pH
Conditioning	5			
Test 3		0.9	0.4	2.0 - 2.3
4		1.2	0.4	1.9 - 2.3
5		1.5	0.4	1.9 - 2.2
6		2.0	0.4	1.9 - 2.1
7		2.5	0.4	1.8 - 2.0
8		1.5	0.4	1.9 - 2.2
9		1.5	0.6	1.9 - 2.2
10		1.5	0.8	1.9 - 2.2
Flotation (Tests 3-10)	3	-	-	2.2 - 2.9

II - Results obtained

Test No.	Al-Fe Conc Weight (%)	Al-Fe Conc Analysis (%) Al_2O_3	Al-Fe Conc Analysis (%) Fe_2O_3	Al-Fe Conc Distribution (%) Al_2O_3	Al-Fe Conc Distribution (%) Fe_2O_3	SiO_2 Conc Analysis (%) Al_2O_3	SiO_2 Conc Analysis (%) Fe_2O_3
3	21.6	9.2	0.53	90.0	55.7	0.28	0.13
4	16.2	12.0	0.75	88.6	52.6	0.30	0.13
5	14.1	13.7	0.77	87.6	47.4	0.32	0.14
6	13.3	14.2	0.76	86.3	43.9	0.37	0.15
7	12.6	14.9	0.86	85.3	46.9	0.37	0.14
8	14.4	13.7	0.88	89.5	55.2	0.27	0.12
9	19.1	10.5	0.76	90.9	63.4	0.25	0.10
10	21.1	9.5	0.70	90.8	64.3	0.23	0.09

Note: The head sample used for these tests analysed 2.2% Al_2O_3 and 0.23% Fe_2O_3 and was used to calculate the analyses of the Al-Fe concentrates.

TABLE 13: Effect of pH on feldspar-iron flotation

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne H_2SO_4	PDD	pH
Conditioning	4			
Test 11		0.95	0.5	2.3 - 2.5
12		1.05	0.5	2.2 - 2.4
13		1.30	0.5	2.1 - 2.3
14		1.50	0.5	1.9 - 2.2
Flotation	3			
Test 11		0.19	-	2.5 - 2.5
12		0.21	-	2.4 - 2.4
13		0.27	-	2.4 - 2.4
14		0.30	-	2.2 - 2.2

II - Results obtained

Test No.	Al-Fe Conc Weight (%)	Analysis (%) Al_2O_3	Fe_2O_3	Quartz*	Distribution (%) Al_2O_3	Fe_2O_3	Quartz**	SiO_2 Conc Analysis (%) Al_2O_3	Fe_2O_3
11	22.2	9.0	0.61	50.3	90.8	61.3	12.7	0.26	0.11
12	18.1	10.9	0.72	40.1	89.9	59.1	8.2	0.27	0.11
13	17.1	11.6	0.75	36.4	89.8	58.6	7.1	0.27	0.11
14	14.1	13.9	0.83	24.1	89.1	53.2	3.8	0.28	0.12

* Calculated as follows: % quartz = 100 - (% Al_2O_3×5.3+% Fe_2O_3+1.4)
** Calculated by difference from 88% quartz in feed

This technique provided more consistent results and enabled the effect of pH to be evaluated more accurately. The feldspar and iron removal were similar, i.e. about 90% and 55%, respectively. The highest concentration of acid tested (Test 14) reduced the quartz floated in the feldspar-iron concentrate from 12.7% to 3.8% without impairing significantly the flotation results. These results compare favorably with those obtained recently by Neal which are listed hereunder (15):

Acidity	Recovery (%)	Lost Quartz (%)
pH 2.7	87.3 feldspar	12.0 quartz
pH 2.6	85.0 feldspar	11.1 quartz
pH 2.5	79.0 feldspar	10.8 quartz
pH 2.3	71.2 feldspar	10.1 quartz
pH 2.2	70.9 feldspar	8.7 quartz

To study the effect of conditioning time on flotation, a series of three tests were conducted under similar conditions except that conditioning time was increased progressively from 3 to 8 min. The details and results of these tests are shown in Table 14.

TABLE 14: Effect of conditioning time on feldspar-iron flotation

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne H_2SO_4	PDD	pH
Conditioning Test				
15	3	1.5	0.4	1.8 - 1.9
16	5	1.5	0.4	1.8 - 1.9
17	8	1.5	0.4	1.8 - 1.9
Flotation	3	-	-	2.3 - 2.4

II - Results obtained

Test No.	Al-Fe Conc Weight (%)	Analysis (%) Al_2O_3	Analysis (%) Fe_2O_3	Distribution (%) Al_2O_3	Distribution (%) Fe_2O_3	SiO_2 Conc Analysis (%) Al_2O_3	SiO_2 Conc Analysis (%) Fe_2O_3
15	19.7	10.3	0.67	92.0	60.0	0.22	0.11
16	14.0	14.0	0.84	89.6	53.3	0.27	0.12
17	11.3	16.7	0.85	85.5	43.6	0.36	0.14

The results of these tests showed that a longer conditioning time improved the selectivity of the flotation but was detrimental to the recovery of feldspar and iron at 8 min. This might be attributed to desorption of the collector.

In one series of tests, the water from the previous test was reused in the next one as a possible means of reducing the reagent and water consumption. Nine cycles of flotation were integrated along the pattern illustrated in Fig. 1.

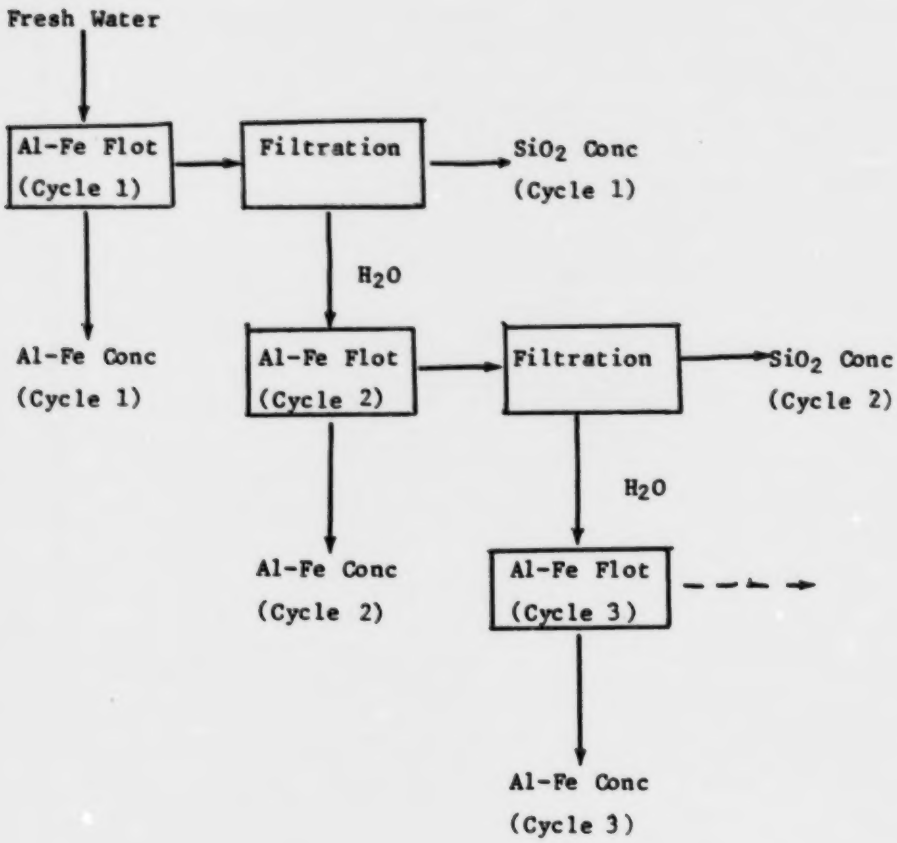

Fig. 1: Illustration of a cycle test

In the early cycles, a slight excess of collector was used, while in the last ones a small shortage was possible without impairing the results as indicated in Table 15.

TABLE 15: Conditions and results of locked cyclical tests

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne H_2SO_4	PDD	pH
Conditioning	5			
Cycle 1		1.5	0.50	1.9 - 2.1
2		1.4	0.47	1.9 - 2.1
3		1.3	0.44	1.9 - 2.1
4		1.2	0.41	2.0 - 2.2
5		1.1	0.38	1.9 - 2.2
6		1.0	0.37	2.1 - 2.3
7		1.0	0.37	2.1 - 2.3
Flotation:	3	-	-	2.3 - 2.5

II - Results obtained

Test No.	Al-Fe Conc Weight (%)	Analysis (%) Al_2O_3	Fe_2O_3	TiO_2	Distribution (%) Al_2O_3	Fe_2O_3	TiO_2	SiO_2 Conc Analysis (%) Al_2O_3	Fe_2O_3	TiO_2
18-1	17.5	11.3	0.65	0.17	89.9	51.6	54.6	0.27	0.13	0.030
18-2	19.8	10.0	0.63	0.15	89.5	56.7	54.4	0.28	0.12	0.031
18-3	18.8	10.7	0.65	0.17	90.5	55.6	57.7	0.26	0.12	0.029
18-4	19.7	9.9	0.63	0.15	88.7	56.4	53.4	0.31	0.12	0.031
18-5	18.5	10.9	0.62	0.16	90.6	52.0	53.2	0.26	0.13	0.032
18-6	18.9	10.7	0.65	0.17	90.9	55.9	58.9	0.25	0.12	0.028
18-7	18.6	10.8	0.57	0.15	90.2	48.2	50.5	0.29	0.14	0.034
18-1 to 7	18.7	10.7	0.63	0.16	90.1	52.7	55.7	0.27	0.13	0.031

Note: Due to moisture left in the cakes, reused acidic water was insufficient. It thus served mostly as make-up water while some fresh water was added in the conditioning stage.

The conditions of the cyclical tests indicated that some economy of reagents and water might be realized by recirculation. However, these were not considerable and the results deteriorated in the 8th and 9th cycles. Therefore, careful testing would be necessary in plant operation before adopting this practice, particularly if the anionic-cationic flotation described later were to be used.

Authors, such as Malghan and Neal, who have studied the non-HF flotation system have concluded that it was less effective for particles over 420 µm. To evaluate the effect of this variable on Magdalen sand, representative samples were cut from the combined flotation concentrates and tailings of two similar and relatively good tests (i.e. Test 2 and 8). Each sample was sized and the fractions weighed and analysed for Al_2O_3 and Fe_2O_3.

TABLE 16: Size analysis of flotation products

Size (µm)	Product	Weight (%)	Analysis (%)		Distribution (%)	
			Al_2O_3	Fe_2O_3	Al_2O_3	Fe_2O_3
+300	Al-Fe conc	2.7	18.3	1.24	85.5	53.4
	SiO_2 conc	32.5	0.26	0.09	14.5	46.7
-300+212	Al-Fe conc	6.4	14.6	0.80	89.4	56.6
	SiO_2 conc	39.3	0.28	0.10	10.6	43.6
-212+150	Al-Fe conc	5.8	8.0	0.78	92.7	77.5
	SiO_2 conc	11.9	0.31	0.11	7.3	22.5
-150	Al-Fe conc	0.8	-	-	-	-
	SiO_2 conc	0.7	-	-	-	-

In the above tests, as well as in a few other tests in which the same calculations were made, an increase in the feldspar and iron recovery was observed in the finer fractions. When considering the grade of feldspar concentrate, a significant decrease from 18.3% Al_2O_3 at +300 µm to 8.0% Al_2O_3 at -212+150 µm was noticed upon sizing. On the basis of this observation a split flotation technique, applicable to this situation, was investigated.

Split flotation technique

In this series of tests, the raw sand was divided into two sized fractions, namely, plus and minus 300 µm, prior to their separate flotation. The coarse fraction floated rather well with minimal amount of reagents (i.e. sulphuric acid and diamine). More difficulty was experienced with the fines, which required more reagents to lower the aluminum and iron to a satisfactory level (see Table 17). No attempt was made to float the coarse fraction at a relatively high pH (i.e. 2.7 to 2.8), or the fine fraction at a low pH (2.1 to 2.2), as recommended by the North Carolina Research Laboratory staff (16).

TABLE 17: Split flotation procedure and results

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne H_2SO_4	Reagents-kg/tonne PDD	pH
Conditioning Test 19 (+300 µm)	5	1.0	0.30	1.8 - 2.1
20 (-300 µm)		1.3	0.45	1.9 - 2.2
21 (-300 µm)		1.3	0.50	1.8 - 2.2
22 (-300 µm)		1.3	0.62	1.9 - 2.1
Flotation (Tests 19-22)	3	-	-	2.5 - 2.7

II - Results obtained

Test No.	Al-Fe Conc Weight (%)	Al-Fe Conc Analysis (%) Al_2O_3	Al-Fe Conc Analysis (%) Fe_2O_3	Al-Fe Conc Distribution (%) Al_2O_3	Al-Fe Conc Distribution (%) Fe_2O_3	SiO_2 Conc Analysis (%) Al_2O_3	SiO_2 Conc Analysis (%) Fe_2O_3
19	11.3	12.5	0.65	84.6	41.1	0.29	0.12
20	13.5	16.0	0.81	87.4	45.8	0.36	0.15
21	17.0	13.1	0.80	89.9	56.7	0.30	0.12
22	27.4	8.2	0.50	90.6	56.8	0.32	0.13
19 to 21 (overall)	15.1	12.8	0.78	87.7	53.6	0.30	0.12

Albeit the results of Tests 20 and 21 indicated that the grade of the fine feldspar-iron flotation concentrate could be improved by a split flotation, the overall results were not encouraging. Also, this approach would require the insertion of an additional screening stage in the flowsheet with a corresponding additional cost.

Anionic-cationic flotation

Various techniques were tested in an attempt to increase iron removal. In experimenting it was found that the addition of high molecular weight petroleum sulphonate (PS) in the flotation circuit achieved interesting results at minimal cost. The simultaneous use of an anionic and a cationic collector has been reported in the literature (17, 18). The conditions and results of three typical tests using this flotation technique are reported in Table 18.

TABLE 18: Effect of reagent concentration on anionic-cationic flotation

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne H_2SO_4	PDD	PS	pH
Conditioning:	5				
Test 23		0.9	0.45	0.37	2.0 - 2.2
24		0.9	0.40	0.30	1.8 - 2.0
25		0.9	0.37	0.26	1.8 - 2.0
Flotation	3	-	-	-	2.3 - 2.5

II - Results obtained

Test No.	Al-Fe Conc Weight (%)	Analysis (%) Al_2O_3	Analysis (%) Fe_2O_3	Distribution (%) Al_2O_3	Distribution (%) Fe_2O_3	SiO_2 Conc Analysis (%) Al_2O_3	Analysis (%) Fe_2O_3	Analysis (%) TiO_2
23	18.8	10.8	0.80	92.3	68.6	0.21	0.086	0.023
24	17.4	11.7	0.81	92.1	64.1	0.21	0.096	0.028
25	15.6	12.7	0.88	89.7	62.4	0.27	0.098	0.028
23 to 25 (overall)	17.3	11.6	0.83	91.5	65.2	0.23	0.093	0.027

In addition to improving the removal of iron and titanium, the petroleum sulphonate reduces the demand for diamine, thus adding very little to the total reagent cost. This might be attributed to a combined action of the two collectors, one helping the other.

Effect of attrition and magnetic separation prior to flotation

Further attempts were made to improve the flotation results and/or reduce the reagent consumption by both an attrition scrubbing and a magnetic separation pre-treatment. The first series of tests consisted of alkali scrubbing at increasing caustic concentration (1.5 to 4.5 kg/t) for periods varying from 5 to 20 min. The slimes produced after attrition were removed by diluting the pulp and decanting rapidly. The most satisfactory conditions were obtained after a 10 to 15 min scrub at 78% solids pulp density and an addition of 2.3 kg NaOH/t. The slimes collected amounted to 0.4 to 0.6% by weight with an average analysis of 4.7% Fe_2O_3 and 7.6% Al_2O_3. This corresponds to some 10% of the original iron and to 2% of the alumina.

The 15 min - 2.3 kg/t conditions were used prior to the flotation experiments. In these tests the total anionic and cationic reagents were progressively decreased from 0.67 kg/t and 0.40 kg/t respectively. It was found that the petroleum sulphonate was almost unnecessary after the alkali scrubbing. Moreover, the results deteriorated when the ratio of diamine to sulphonate was less than about 4:1 as indicated by the following results.

TABLE 19: Effect of alkali scrubbing and desliming

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne NaOH	H_2SO_4	PDD	PS	pH
Scrubbing (desliming 2x)	15	5	-	-	-	
Conditioning	5					
Test 26			1.50	0.40	0.27	1.9 - 2.1
27			1.50	0.40	0.10	2.0 - 2.2
28			1.50	0.40	-	2.0 - 2.2
Flotation	3	-	-	-	-	2.4 - 2.6

II - Results obtained

Test No.	Al-Fe Conc* Weight (%)	Analysis (%) Al_2O_3	Analysis (%) Fe_2O_3	Distribution (%) Al_2O_3	Distribution (%) Fe_2O_3	SiO_2 Conc Analysis (%) Al_2O_3	Analysis (%) Fe_2O_3	Analysis (%) TiO_2
26	16.8	12.8	0.92	91.8	70.4	0.23	0.078	0.022
27	17.3	11.7	0.91	91.8	71.4	0.22	0.076	0.021
28	17.6	10.8	0.88	86.3	70.1	0.38	0.080	0.021

*Including the slimes from attrition.

If we compare the data of Test 27 to that of Test 24, we find that for a similar weight of float product, e.g., 17.4 versus 17.3%, the alkali scrubbing and desliming has resulted in improving the iron flotation by 7.3%, while reducing the petroleum sulphonate consumption by 0.2 kg/t. However, these benefits are low when compared to the high energy and water costs involved in attrition.

By employing magnetic separation as pre-treatment, some 50% of the iron could be removed prior to flotation. The latter did not require any petroleum sulphonate and lowered the iron content of the silica concentrate to 0.06% Fe_2O_3 in the best test which is shown by the results of Table 20.

TABLE 20: Magnetic separation and feldspar-iron flotation (Test 29)

I - Conditions

Magnetic Separation:	Flotation:
Sala HGMS	5-min conditioning
6.8 kilogauss	0.30 kg PDD/tonne
55.5 mm/sec	pH-2.20 to 2.35

II - Results obtained

Operation	Products	Weight (%)	Analysis (%)		Distribution (%)	
			Al_2O_3	Fe_2O_3	Al_2O_3	Fe_2O_3
Magnetic Separation	Mags	6.9	4.45	1.59	13.9	53.9
	Non-mags	93.1	2.03	0.10	86.1	47.1
	Feed	100.0	2.20	0.20	100.0	100.0
Flotation	Al-Fe conc	14.7	11.7	0.31	78.3	23.0
	SiO_2 conc	78.4	0.22	0.061	7.8	24.1
	Feed	93.1	2.02	0.10	86.1	47.1

This approach was abandoned because of high silica and feldspar losses in the magnetic fraction and the additional cost of the treatment.

Mineralogy of Selected Flotation Products

Representative samples of the silica and feldspar concentrates from Tests 23 to 27 were submitted to the Mineralogy Section of CANMET for identificaion of the principal minerals. A summary of the observations follows (19).

The silica concentrate contained quartz with little alkali feldspars and only traces of rutile and chlorite. Some of the quartz grains are coated or intergrown with a mixture of an iron-rich clay and an iron oxide mineral, likely limonite.

The feldspar concentrate was sized into four main fractions prior to examination. The following minerals, listed in their approximate order of decreasing abundance, were identified: K-feldspar, quartz, plagioclase,

minor amounts of ilmemite and rutile, traces of chlorite, apatite, biotite, hematite and Cr-Al-Fe spinel. The K-feldspar is highest in the coarse-grained fractions (+210 µm) and low in the finer ones (see Fig. 2 and 3). The hematite, ilmenite, rutile and spinel are present mainly in the minus 150 µm fractions,as shown in Fig. 3 and 4. Their content increases with the fineness of the grains.

To prepare a better sample for identification of the iron-bearing minerals, a portion of the quartz concentrate and one of the feldspar products were separately treated on a Stearns laboratory high-intensity magnetic separator at a setting of 5000 gauss. Very few magnetics (0.65% by weight) were obtained from the quartz concentrate. In the feldspar product, the magnetic fraction attained 5.5% by weight thus indicating that some 60% of the iron minerals (on a weight basis) had floated. The two magnetic fractions were combined for mineralogical examination. This examination indicated the presence of an iron-clay mineral, chlorite, biotite, pyroxene, and less of rutile, ilmenite, hematite and pyrite along with quartz and feldspars. The quartz was often intergrown with the iron oxides and the Fe-rich clay mineral.

Purification of Silica Concentrate

Acid leaching

Acid leaching was a logical approach to eliminate iron oxide coating on the quartz grains to make the silica sand suitable for flint glass manufacture. Two acids, sulphuric and hydrochloric, were tested in this regard. The rate of iron dissolution was dependant on the acid concentration, contact time, and leach temperature. The details of the hydrometallurgy testwork are given in a separate report (20). Only a summary of the most significant results will be given here. For instance the set of curves shown in Fig. 6 are highly illustrative of the iron rate of dissolution. With the concentrated hydrochloric acid, the iron oxide level was lowered to 0.017% after 2 h and 0.012% in 6 h. When sulphuric acid was used it took about 4 h to attain 0.035% Fe_2O_3.

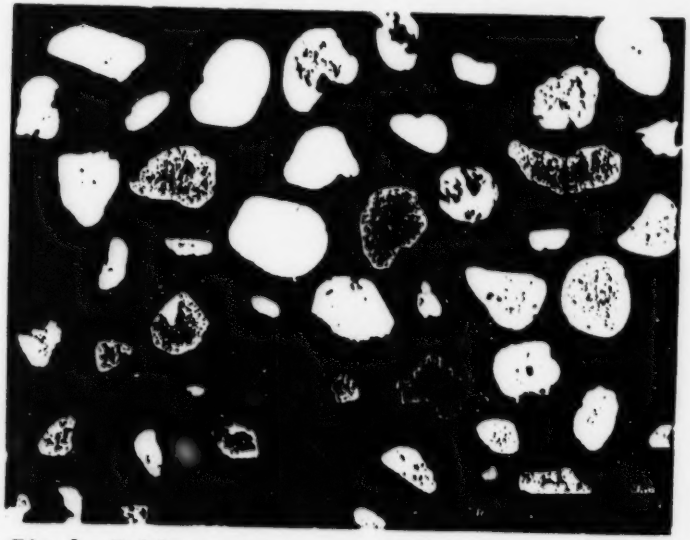

Fig 2. Feldspar-iron conc (+420 µm)

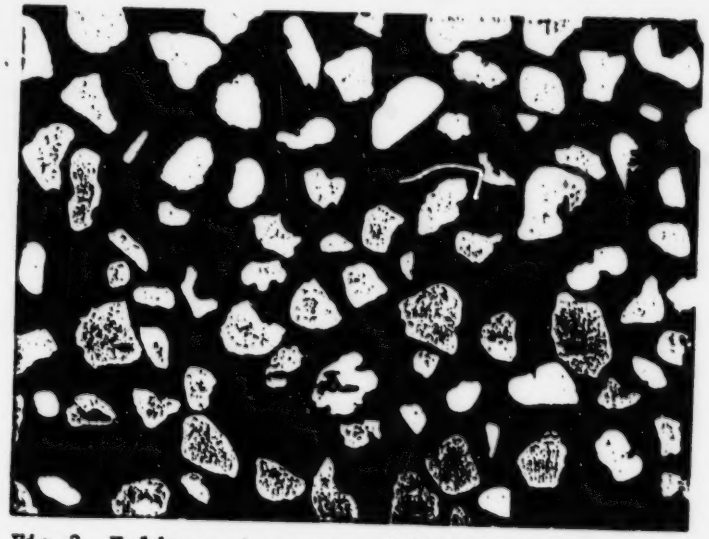

Fig 3. Feldspar-iron conc (-420+210 µm)

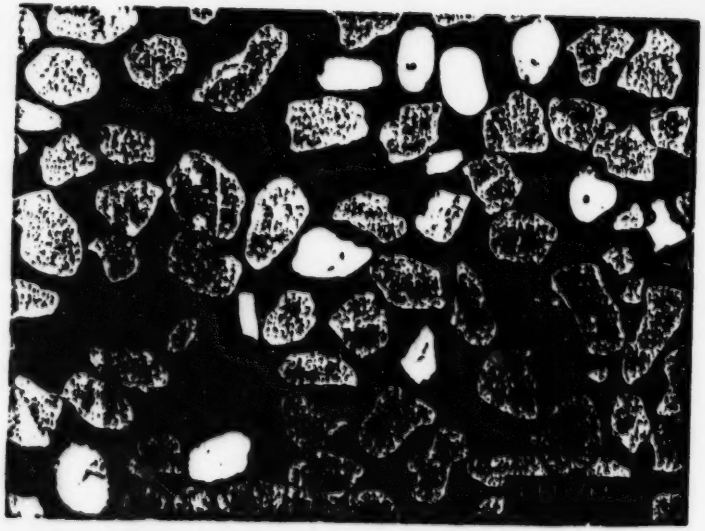

Fig 4. Feldspar-iron conc (-149+105 µm)

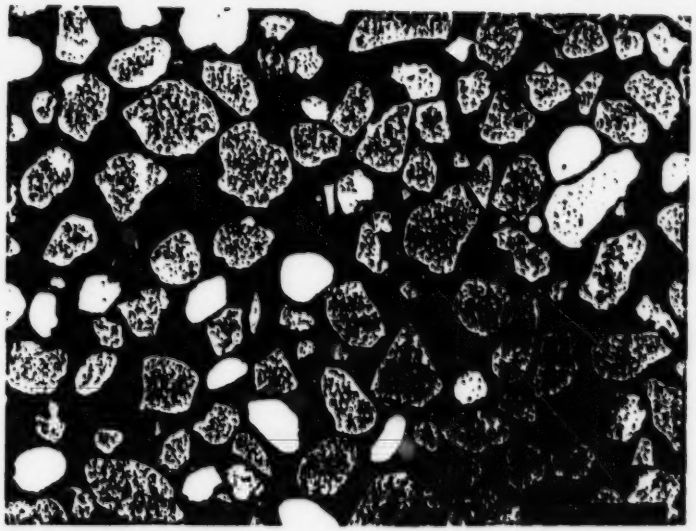

Fig 5. Feldspar-iron conc (-105 µm)

Legend: The dark grey grains are quartz, the light grey ones are feldspar and the white particles are ilmenite, rutile and spinel.

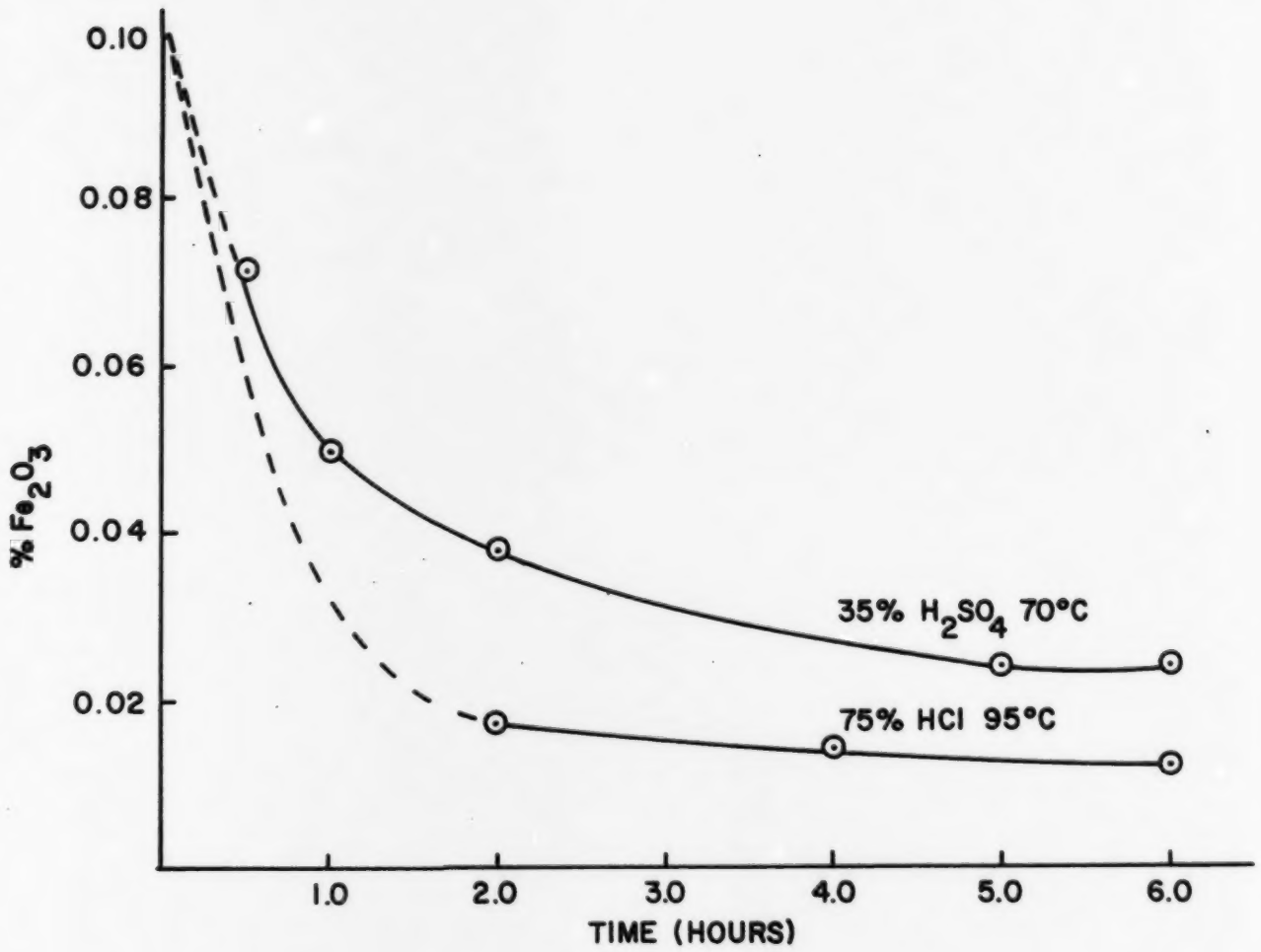

FIG. 6-ACID EXTRACTION OF IRON AS FUNCTION OF TIME AT 1:1 SOLID-LIQUID RATIO

Although the hydrochloric acid gave better results, the sulphuric acid was selected for further testwork because of its availability and much lower cost near Gaspé. To render the process economically attractive, it would be imperative to recycle the acid several times. To simulate this, a series of leaching tests were made in which the acid from the one test was used in the next. The same acid was reused up to 10 times in this manner without too much detrimental effect on the iron extraction as indicated by the following results. The silica concentrate used for the multiple leaching was a blend from Tests 2 to 27 which analysed 0.11% Fe_2O_3.

TABLE 21: Results of H_2SO_4 recycled leaching (Test 30)
(35% H_2SO_4 at 1:1 and 70°C for 6 h)

Leach Cycle	Leached SiO_2 Conc % Fe_2O_3	Leach Cycle	Leached SiO_2 Conc % Fe_2O_3
1	0.037	6	0.050
2	0.041	7	0.044
3	0.037	8	0.044
4	0.044	9	0.047
5	0.036	10	0.040
Average	0.039	Average	0.045

The iron content of these leach residues was considered slightly high at 0.039 to 0.045%. To further reduce the iron oxide level to 0.035%, three methods were investigated:

a) The first method consisted of screening the minus 180 µm from the leach products.

b) An alkali scrubbing-desliming step after or prior to H_2SO_4 leaching was used in the second approach.

c) This method employed high intensity magnetic separation after acid leaching.

Although these treatment methods were not absolutely necessary in pilot-plant testing for achieving the desired 0.035% Fe_2O_3, it is nonetheless worthwhile to report the laboratory results.

Removal of fines from leach products

Each of the cycled leach products was screened on a 180 µm sieve, with the +180 µm fractions being analysed for iron oxide. This procedure reduced the Fe_2O_3 content by some 17.5% as shown in Table 22.

TABLE 22: Analysis of +180 µm recycled leach products (Test 31)

Leach Cycle	Leached SiO_2 Conc % Fe_2O_3	Leach Cycle	Leached SiO_2 Conc % Fe_2O_3
1	0.036	6	0.044
2	0.035	7	0.031
3	0.032c	8	0.036
4	0.039	9	0.033
5	0.033	10	0.035c
Average	0.035	Average	0.036

c = calculated

Alkali scrubbing-desliming of leach products

A combined sample was prepared from the cycled leach products (0.043% Fe_2O_3) and scrubbed for 15 min at 75% solids with 2.5 kg NaOH/t. The scrubber used was a laboratory 1000 g Wemco unit and the desliming was carried out by decantation from a pail (twice). The results obtained are shown in Table 23.

Since the slimes also contain 3.0% Al_2O_3, it might be inferred that they are composed of the iron-rich clay mineral. The method, albeit producing good results with minimal silica losses, is costly in energy and water requirements. It was decided therefore to apply alkali scrubbing before the leaching stage for possible savings in the acid consumption.

TABLE 23: Results of scrubbing and desliming H_2SO_4-leach residues (Test 32)

Product	Weight (%)	Analysis % Fe_2O_3	Distribution % Fe_2O_3
SiO_2 Conc	99.7	0.032	73.7
Slimes	0.3	3.76	26.3
Feed	100.0	0.043	100.0

Attrition and sulphuric acid leach

In this experiment, a mixed quartz concentrate from the flotation Tests 2 to 27 was scrubbed and deslimed prior to a series of sulphuric acid leach tests. The conditions of attrition and leaching were as follows:

Scrubbing	H_2SO_4-Leach
5 min	6 h
900 rpm	70 °C
75% solids	50% solids
2.3 kg NaOH/t	35% H_2SO_4 (vol)
Desliming (x)	15 cycles

The acid was recycled 15 times to check for saturation of solution. Although the deslimed quartz concentrate still contained 0.10% Fe_2O_3, the scrubbing operation was considered beneficial to the subsequent leaching operation as shown by the following results.

TABLE 24: Results of H_2SO_4 recycled leaching after alkali scrubbing and desliming (Test 32)

Leach Cycle	Deslimed & Leached SiO_2 Conc (% Fe_2O_3)	Leach Cycle	Deslimed & Leached SiO_2 Conc (% Fe_2O_3)	Leach Cycle	Deslimed & Leached SiO_2 Conc (% Fe_2O_3)
1	0.031	6	0.036	11	0.040
2	0.022	7	0.032	12	0.043
3	0.039	8	0.035	13	0.024
4	0.034	9	0.035	14	0.026
5	0.023	10	0.026	15	0.027
Average	0.030	Average	0.033	Average	0.032

These results show that attrition treatment improves the iron extraction significantly. The acid solution was maintained effective for 15 cycles which is important when considering possible savings in a plant operation.

Upgrading of Feldspar Concentrate

To be of commercial value the feldspar product must attain at least 15% Al_2O_3. Mineralogical examination of the rougher feldspar-iron flotation concentrates has indicated that a high proportion of free quartz was still present in this product. The first procedure tested to upgrade the feldspar concentrate was thus a cleaner flotation stage. Three approaches were investigated for this operation. In the first, no additional reagent was used. In the second, a small addition of sulphuric acid was made to maintain a low pH and some collector was added to prevent any loss of feldspar. Finally, a high density conditioning was carried out using both sulphuric acid and one or two collectors. The details of five typical tests and their results are shown in Table 25. The cleaner flotation tests were all conducted on 200 g samples obtained from the overall feldspar-iron concentrate of Test 18, which analysed 10.7% Al_2O_3, 0.63% Fe_2O_3 and 0.16% TiO_2.

TABLE 25: Conditions and results of feldspar-iron cleaner flotation

I - Reagents and conditions

Operation	Time min	Reagents-kg/tonne H_2SO_4	PDD	PS	pH
Conditioning					
Test 33-34	nil	-	-	-	-
35	3	0.20	0.05	-	2.0 - 2.2
36	3	0.25	0.05	-	1.9 - 2.1
37	3	0.20	0.08	0.02	2.1 - 2.2
Flotation					
Test 33	3	-	-	-	2.5 - 2.8
34	3	0.15	0.03	-	2.3 - 2.4
35-37	3	-	-	-	2.3 - 2.4

II - Results obtained

Test No.	Al-Fe Cleaner Conc Weight (%)	Analysis (%) Al_2O_3	Analysis (%) Fe_2O_3	Distribution (%) Al_2O_3	Distribution (%) Fe_2O_3	Cleaner Tailing Analysis (%) Al_2O_3	Fe_2O_3	TiO_2
33	73.5	14.1	0.70	96.8	81.5	1.3	0.42	0.09
34	75.2	14.0	0.73	98.2	86.7	0.8	0.34	0.07
35	69.4	15.0	0.73	97.3	80.2	1.0	0.41	0.08
36	67.7	15.1	0.75	95.8	80.5	1.4	0.38	0.07
37	65.3	15.9	0.74	97.4	76.3	0.8	0.46	0.10

To obtain a 15% Al_2O_3 concentrate, it was necessary to repulp and condition at high pulp density (65% solids) with the pH maintained at 1.9-2.2. Test 37 which used the anionic-cationic system gave the best overall results. In all tests, it was observed that the iron and titanium oxide fell significantly in the cleaning stage. Further testwork on feldspar purification will be carried out in the near future and reported separately.

PILOT-PLANT TESTWORK

Outline

The object of the pilot-plant testwork was principally to produce some 200 kg of clear grade glass sand and 300 kg of steel foundry sand. The procedure adopted to obtain the glass sand consisted essentially of feldspar-iron flotation (employing the optimum conditions previously found) followed by sulphuric acid leaching. Acid leaching was not necessary for the production of foundry sand, but limited classification was required to obtain an average size comparable to that of Ottawa sand. In addition, attempts were made to reduce the consumption of flotation reagents to a minimum without impairing the results.

The production of both glass and foundry sands required four main unit operations, namely:

a) coarse and fine screening to remove sea shells, highly contaminated fines, and salt water;
b) high density acidic conditioning prior to flotation for optimizing reagent adsorption;
c) rougher and cleaner flotation to separate the feldspar and iron minerals from the quartz and to upgrade the feldspar concentrate;
d) size classification for the foundry sand and acid leaching for the glass sand.

Equipment and Flowsheet

The following pilot-plant equipment was used:

- a 0.8 m diameter Sweco screen with a 0.5-mm opening for the removal of the sea shells;
- a similar sieve with a 0.18-mm opening for screening out the barren fines;
- a 0.3-m diameter conditioner equipped with two baffles, an inlet feed pipe and multiple overflow outlets. (The shaft had two straight blades and rotated at 900 rpm under load. A more conventional unit with a four inclined-blade agitator, turning at 600 to 700 rpm, would have been more effective);
- three 2-L, acid resistant rougher flotation cells installed in series;

- the same conditioner and two 1-L (equivalent capacity) cells were used for the cleaner flotation;
- a leaching vessel with a 13.5-kg (solid) capacity.

A flowsheet illustrating the assembly of this equipment is shown in Fig. 7. It should be noted that no pumping was used in the flotation circuit since all pulp transporatation was made by gravity.

Operation and Control

Since the screening capacity considerably exceeded that of the flotation circuit, the two pre-sizing operations were carried out prior to initiating flotation, the screened products being stored wet. It was found necessary, since plant sieving is not an effective operation, to repass the coarse oversize product so that excessive losses of misplaced sand with the sea shells could be avoided. For fine screening, an 180-µm sieve was used to obtain a desirable separation at about 150 µm, with the minus material being discarded due to a high impurity level.

Flotation forms the basis of the whole process and thus required careful control. The critical factors, namely, pH, pulp density and reagent addition were recorded every 15 min and kept as constant as feasible during each mill run. To stabilize the pH as much as possible, all sulphuric acid used in the circuit was added as acidified water. The acid required for the conditioning stage was maintained at pH 1.75 to 1.80, while dilution water into the flotation circuit was kept at pH 2.35 to 2.45.

Rougher and cleaner flotation were run separately and not consecutively due to shortage of equipment (e.g., conditioner). The results of the two operations, however, were integrated in the material balances for simplicity and completeness.

In respect to acid leaching, a continuous acid leaching apparatus consisting of a small thickener equipped with a recirculating device was initially tested, but pumping difficulties were encountered and this approach was abandoned. It was replaced by a series of 13.5-kg batch tests. These had the advantage of permitting the investigation of multiple reutilization of the sulphuric acid liquor.

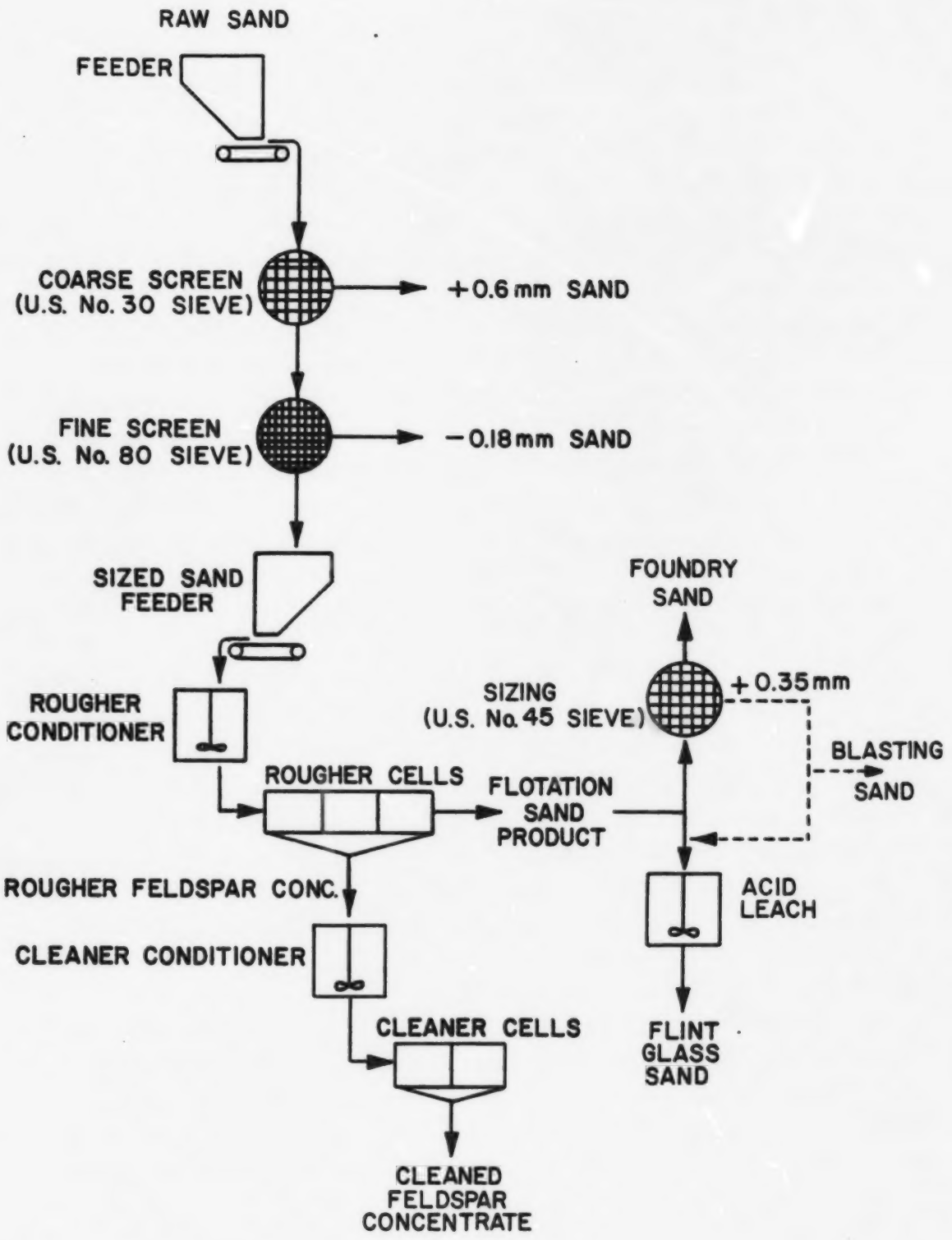

FIG. 7 – PILOT PLANT FLOWSHEET FOR MAGDALENE SAND BENFICIATION

To improve the size of sand for foundry manufacture, some plus 350 µm material was screened from the silica flotation concentrates. Further improvement was obtained by adding the finest size sand from the silica glass sand, to the foundry sand. With these two operations, the grade of the glass sand was improved prior to leaching and the fineness of the foundry sand was comparable to Ottawa sand (i.e., AFS No. 52). In a plant operation, the sand should, however, be classified by hydro-sizing for greater flexibility.

Head Sampling and Analysis

A 2-t sample of wet raw sand was received in 96 fibreglass bags for the pilot plant work. Since the material was not dried, it was necessary to determine the moisture and soluble content by other standard methods. These levels were established at 9.5% and 0.35%, respectively. The soluble matter was most likely sodium chloride from sea water. Although sodium chloride is generally not thought harmful to flotation, it was thought advisable to remove it by washing. This was accomplished during the fine screening stage. Fifty-four bags of raw sand were picked at random from the shipment and blended in a cement mixer. After thorough mixing a head sample was riffled out of the mixed product for chemical analysis. In addition to analysing a bulk head sample, it was decided to analyse a sized head sample from which the +590 µm and the -149 µm material had been removed, since the material being fed to the flotation circuit was essentially -590+149 µm. The analyses of the unsized and sized sand fractions are presented in Table 26. The waste product analyses were calculated by difference.

Preliminary Mill Tests

Prior to undertaking the production of the glass and foundry sand, it was decided to re-study briefly the main variables of the flotation system on the pilot-plant scale. Short runs, referred to as mill tests, were thus carried out where the conditioning time and the concentration of reagent(s) were varied while maintaining the pH as constant as possible. Each set of conditions was experimented for about 1 h, of which the last 20 to 40 min were sampled. For this initial pilot-plant work, only the pre-screening and the rougher feldspar-iron flotation circuits were operated. The coarse and fine screened waste products accounted for only 2% of the feed weight and were not analysed. The details of the individual tests and the results obtained are reported in Table 27.

TABLE 26: Analysis of pilot-plant head samples

Product	Weight (%)	Analysis (%)								
		SiO_2	Al_2O_3	K_2O	Na_2O	CaO	MgO	Fe_2O_3	TiO_2	LOI
Raw Sand*	100.0	95.0	2.16	1.13	0.30	0.16	0.15	0.23	0.05	0.4
Sized Sand	98.9	95.8	2.06	1.10	0.27	0.13	0.07	0.18	0.04	0.3
Waste ** (coarse and fine)	1.1	23.6	11.2	3.8	3.0	2.8	7.3	4.7	1.2	-

*The following trace elements were also determined by Pilkington Brothers (England) Ltd:

C	ZrO_2	Chloride	Mn	Ni	Cr	Cu	V	Co
0.04%	0.02%	110 ppm	25 ppm	22 ppm	2 ppm	2 ppm	<2 ppm	<1 ppm

** Composed of 0.2% coarse waste (sea shells) and 0.9% of -150 µm fines. Some of the calculated analyses appear high, e.g., Al_2O_3, Na_2O and MgO.

TABLE 27: Pilot plant feldspar-iron flotation at various conditioning times and reagents concentration

I - Reagents and conditions

Constant	Variables: Conditioning Time (min)	Variables: Reagents kg/tonne PDD	Variables: Reagents kg/tonne PS	Mill Test: Series	Mill Test: Number
I-Feed rate:	5	0.45	-	A	1
70 kg/h	8	"	-		2
(1.98% Al_2O_3,	10	"	-		3
0.17% Fe_2O_3)					
II-Conditioning:	8	0.39	-	B	1
1) 57±1% solids	"	0.42	-		2
2) pH_i = 1.8±0.05	"	0.45	-		3
3) pH_f = 2.2±0.1	"	0.51	-		4
III-Flotation:	"	0.32	0.10	C	1
1) 27±2% solids	"	0.39	0.10		2
2) pH_i = 2.3±0.1	"	0.42	0.10		3
3) pH_f = 2.35±0.1	"	0.45	0.10		4

II - Results obtained

Mill Test	Weight* (%)	Al-Fe Conc: Analysis (%) Al_2O_3	Fe_2O_3	Quartz**	Al-Fe Conc: Distribution (%) Al_2O_3	Fe_2O_3	Quartz	SiO_2 Conc: Analysis (%) Al_2O_3	Fe_2O_3	Quartz**
A-1	13.1	13.3	0.52	27.5	87.7	35.9	4.1	0.28	0.14	98.2
A-2	15.4	11.4	0.57	37.5	89.6	48.6	6.5	0.24	0.11	98.5
A-3	15.1	11.7	0.58	35.9	91.2	50.8	6.1	0.20	0.10	98.7
B-1	12.5	14.1	0.52	23.3	88.9	38.3	3.3	0.26	0.12	98.4
B-2	14.2	12.6	0.54	31.8	90.5	45.0	5.1	0.22	0.11	98.6
B-3	15.7	12.0	0.53	34.4	91.4	49.7	6.1	0.21	0.10	98.6
B-4	16.7	10.9	0.54	40.2	92.0	53.3	7.5	0.19	0.095	98.7
C-1	11.0	15.6	0.65	15.2	87.7	38.2	1.9	0.27	0.13	97.7
C-2	13.5	13.3	0.62	27.4	89.6	46.8	4.2	0.24	0.11	98.4
C-3	14.0	12.1	0.59	33.8	90.0	49.0	5.3	0.22	0.10	98.6
C-4	16.2	11.3	0.58	38.0	91.7	54.1	6.9	0.20	0.095	98.7

* Calculated algebraically using the three-product formula.

** Calculated as follows: Fe-Al conc: % quartz = 100 - (%Al_2O_3×5.3 + %Fe_2O_3+1.5); SiO_2 conc: % quartz = 100-(%Al_2O_3×5.3+%Fe_2O_3+0.15)

The conclusion of these mill tests was that slightly less reagents, but longer conditioning time, were required to obtain results comparable to those in the laboratory. In the pilot plant, conditioning time was calculated as the total retention period in the conditioner and thus assumes perfect mixing.

At the request of Magdalen Silica Inc. a mill test was carried out without fine screening at 180 µm. The constant conditions of Table 27 were used with an 8-min conditioning time and a high reagent addition, i.e. 0.48 kg diamine dioleate and 0.10 kg petroleum sulphonate per tonne of feed. The results obtained were relatively good with a feldspar-iron concentrate containing 14.4% Al_2O_3 and 0.61% Fe_2O_3 and a silica concentrate analysing 0.26% Al_2O_3 and 0.11% Fe_2O_3, but somewhat inferior to those obtained when using fine screening (see Table 27). The unfloated minus 180 µm material was rather contaminated analysing 0.45% Al_2O_3 and 0.29% Fe_2O_3.

The criteria for selecting the conditions of the major mill runs were as follows:

a) for foundry sand the Al_2O_3 content shall not exceed 0.25% (as recommended by Dr. Szabo);

b) for glass sand Al_2O_3 and Fe_2O_3 were to be kept at about 0.2% and 0.1%, respectively;

c) the reagent consumption should be as low as possible;

d) the selectivity of the quartz separation from the feldspar and iron minerals should be as high as possible.

The latter is evaluated using a composite selectivity index (S.I.) formulated as follows:

$$+S.I. = \sqrt{\frac{Qc\ (Fc+Ic)}{Qr\ (Fr+Ir)}}$$

where Qc, Fc and Ic are the quartz, feldspar and iron oxide recovered in their respective concentrates and Qr, Fr and Ir are the quartz, feldspar and iron not reporting in their own concentrates. Selectivity indices were computed for Tests B-2 to 4 and C-2 to 4, which met either criteria (a) or (b). The values obtained are compared in Table 28 with other data pertinent to the selection of the parameters for the mill runs.

TABLE 28: Collector(s) consumption, significant results and selectivity indices of mill tests

Data		B-1	B-2	B-3	C-1	C-2	C-3
Collector(s)		0.42	0.45	0.51	0.49	0.52	0.55
SiO_2 Conc:	$\%Al_2O_3$	0.22	0.21	0.19	0.24	0.22	0.20
	$\%Fe_2O_3$	0.11	0.10	0.095	0.11	0.10	0.095
Selectivity Indices		6.25	6.06	5.72	6.93	6.30	6.02

After analysing these data and results, it was decided to use the conditions of Tests B2 for the production of the foundry sand and of Test C-4 for the production of the glass sand.

Mill Run I (Glass Sand Production)

Flotation conditions and results

The first major mill run was oriented towards the production of silica sand for the glassmaking industry. The main feature of the test was the utilization of the two-collector system to reduce both the feldspar and iron to minimum levels. This approach proved in the laboratory to be the most satisfactory for achieving good results with a limited equipment requirement (i.e. no attrition, no magnetic separation, etc.).

The mill run was sampled for 4.5 h and all products were kept. Since it is a pre-requisite of the glass industry that the raw sand be chemically constant, the silica flotation product was sampled for 20 sec every 10 min. By sampling with this frequency and analysing composite samples representing each half hour of production, the chemical consistency could be verified. The rougher feldspar-iron flotation concentrate was also sampled similarly. In addition, the quartz bulk concentrate of every 1.5 h was riffled by conventional means and analysed. All these analyses are reported in Appendix I and averaged for the calculation of the material balance. Samples of the other products of the circuit, e.g., flotation feed and feldspar-iron cleaner tailing, were riffled for analysis at the end of the mill run. The coarse and fine wastes were analysed only once during the total pilot-plant work.

Other pertinent data on Mill Run I and a material balance are given in Table 29.

TABLE 29: Data for Mill Run I (glass sand production)

I - Flotation conditions and reagents

(Feed rate: 70 kg/h, dry basis)

Operation	Time min	Dilution % solids	Reagent kg/tonne H_2SO_4	PDD	PS	pH_i	pH_f
Rougher circuit:							
Conditioning	8	56 - 61	1.0	0.45	0.10	1.8	2.2-2.3
Flotation	1.8	28_i-24_f	0.7	-	-	2.2-2.4	2.3-2.4
Cleaner circuit:							
Conditioning	4	60 - 62	0.05	0.03	0.01	2.2-2.3	2.3-2.4
Flotation	0.7	15_i-6_f	0.35	-	-	2.3-2.4	2.3-2.4

II - Material balance

Products	Weight (%)	Analysis (%) Al_2O_3	Fe_2O_3	Quartz*	Distribution (%) Al_2O_3	Fe_2O_3	Quartz
SiO_2 conc	81.4	0.22	0.10	98.7	8.7	40.4	90.9
Al-Fe cl conc	11.1	14.92	0.62	18.9	80.2	33.8	2.4
Al-Fe cl tail	4.8	2.51	0.55	85.7	5.8	13.2	4.7
Coarse waste	2.2**	4.16	0.67	65.0	4.4	7.3	1.6
Fine waste	0.5	3.64	2.08	77.0	0.9	5.3	0.4
Feed	100.0	2.06	0.20	88.4	100.0	100.0	100.0

Symbols: $_i$ = initial and $_f$ = final

* Calculated

** Composed of 0.2% sea shell, 0.1% oversize sand and 1.9% misplaced grains.

Post-screening and acid leaching

The silica sand produced in Mill Run I was screened on a 210 µm sieve. The purpose of this operation was to improve the grade of the sand to be leached (as demonstrated by the laboratory tests) and by transferring some fines to the foundry sand, to increase this sand AFS Grain Fineness Number. The efficiency of screening the silica sand, however, was rather poor because the fine grains, still coated by collectors, had a strong tendency to adhere to the larger particles, which resulted in fines reporting with the oversize fraction. A sample of sand was re-screened a second time on a dry vibrating unit, but little additional undersize was removed as shown in Table 30.

TABLE 30: Results of sizing prior to leaching

Product	Weight (%)	Analysis (%)			Distribution (%)		
		Al_2O_3	Fe_2O_3	TiO_2	Al_2O_3	Fe_2O_3	TiO_2
+210 µm Sand (screened twice)	92.6	0.19	0.095	0.030	87.4	85.9	88.2
-210 µm Sand	7.4	0.36	0.20	0.050	12.6	14.1	10.8
Feed*	100.0	0.20	0.103	0.031	100.0	100.0	100.0
+210 µm Sand (screened once)	95.1	0.22	0.102	0.027	91.4	90.9	90.7
-210 µm Sand	4.9	0.40	0.20	0.053	8.6	9.1	9.3
Feed	100.0	0.23	0.107	0.028	100.0	100.0	100.0

* The material which was screened twice corresponds essentially to the first 1.5 h of silica flotation production (see Appendix I).

The two samples of "coarse" sand (i.e., screened and re-screened) were acid leached employing the conditions reported in Table 31. Three batches of the re-screened sand, designated A, B and C, were leached using

fresh acid; two batches of the single-screened sand, designated D and E, were also leached with fresh acid. In addition 12 batches of single-screened sample were prepared and a test was conducted in which the acid from the preceeding batch was reused in the succeeding batch. These batches were identified as leach cycles I to XII. The results with the double-screened sand were slightly better than the results obtained with the single-screened material, but the results did not deteriorate appreciably in the twelve cycles of acid reutilization (see Table 31). Six hours of sulphuric acid leaching were used in all the above tests. A control leach, however, in which aliquot samples were cut and analysed every hour, suggested that 4-h would be sufficient to attain the desired 0.035% Fe_2O_3. This confirmed a similar finding on the laboratory scale.

During the pilot-plant study, several glass manufacturers visited CANMET and observed the work in progress. At the time of the visit only leached batches A, B and C were completed. A combined sample from these was provided to the visitors. Some subsequently reported that this silica product was of sufficient quality for the manufacture of clear glass. One major manufacturer carried out a complete analysis of this sand. Results are reported in Table 32 with some comparative CANMET analyses. The two sets of analyses are in relatively good agreement.

TABLE 31: Summary of pilot-plant acid leaching
(35% H_2SO_4 by vol, 50% solids, 70°C)

Leach Feed	Weight-kg (dry basis)	Leaching Time h	Leach Sample Identification	Glass Sand Analysis (%) Fe_2O_3	Glass Sand Analysis (%) Al_2O_3	Iron Extraction* (%)
Silica Sand +210 µm (screened twice)	13.5	6	Batch A	0.032	0.22	66.3
	13.5	6	" B	0.033	0.19	65.3
	13.5	6	" C	0.031	0.18	67.4
Silica Sand +210 µm (screened once)	27.0	6	Batch D & E	0.034	0.25	66.7
	13.4	6	Cycle** I	0.031		69.6
	13.4	6	" II	0.033	0.19	67.6
	14.2	6	" III	0.042		58.2
	13.4	6	" IV	0.036	0.23	64.7
	13.6	6	" V	0.037		63.8
	12.8	6	" VI	0.037	0.19	63.8
	13.6	6	" VII	0.035	0.18	65.7
	13.6	6	" VIII	0.029	0.19	71.6
	13.6	6	" IX	0.041	0.18	59.8
	12.8	6	" X	0.029	0.20	71.6
	13.7	6	" XI	0.028	0.17	72.4
	13.0	6	" XII	0.042	0.24	58.2
	13.5	1	Leach Res -1 h	0.047	0.18	53.9
	13.5	2	" " -2 h	0.036	0.16	64.7
	13.5	3	" " -3 h	0.037	0.18	63.8
	13.5	4	" " -4 h	0.033	0.17	67.6
	13.5	5	" " -5 h	0.035	0.17	65.7
	13.5	6	" " -6 h	0.034	0.18	66.7
TOTAL	242.1	6	Glass Sand Composite	0.034	0.20	66.3

* Calculated by difference from 0.095% Fe_2O_3 for Batches A, B and C, and from 0.102% Fe_2O_3 for all the others.

** In the cycle-leach tests the Fe_2O_3 analyses were made by wet chemical methods whereas the Al_2O_3 was determined by optical emission spectrometry.

TABLE 32: Analysis of typical glass sand

Laboratory	SiO_2(%)	Al_2O_3(%)	K_2O(%)	Na_2O(%)	MgO(%)	CaO(%)	Fe_2O_3(%)	TiO_2(%)
Pilkington*	99.6	0.20	0.07	<0.03	0.05	0.01	0.025	0.02
CANMET	99.6	0.16	0.06	<0.03	<0.1	<0.1	0.027	0.023

	LOI(%)	C (%)	ZrO_2(%)	Chloride	Mn	Cr_2O_3	V	Ni,Cu,Co
Pilkington*	0.1%	0.02%	<0.01%	30 ppm	4 ppm	2 ppm	<2 ppm	<1 ppm

* Pilkington Brother Ltd, R&D, Latham, England, through the courtesy of Mr. J.D. Greenall, Pilkington Brothers Canada Limited.

Since this sample represented some 40 kg of the best glass sand produced, with only 0.025 to 0.027% Fe_2O_3 and a minimal amount of other metallic impurities, initial attempts were made to upgrade it to optical and glassware requirements (e.g. Fe_2O_3 0.008 to 0.013%) by either magnetic separation or hydrochloric acid leaching. At present, the magnetic separation approach appears more encouraging with two types of separators, namely the Cryogenic and the Sala HGMS (high gradient), which reduced the iron oxide to about 0.015%. Further work supported by very accurate analyses would be required to draw conclusions on this point.

Mill Run II (Foundry Sand Production)

Flotation and classification

This pilot-plant run was generally similar to Mill Run I, except that only one collector (dioleate) was used in the feldspar-iron rougher flotation stage and none in the cleaner stage. Previous testwork had indicated that the anionic collector is not necessary for the production of foundry sand as the iron content is not critical. The leaching operation was omitted for the same reason.

Mill Run II lasted 6.5 h and was spread over two days. Timed and bulk samples of the flotation circuit were cut along the pattern set in the first run. A flotation head sample was also cut at regular intervals, but analysed only once as were the other end products. The individual analyses are given in Appendix II, while details on the reagents and conditions are shown in Table 33 along with the overall material balance.

A standard screen test was made on a representative sample of the total "foundry" sand produced and the AFS Grain Fineness Number calculated at 49. The addition of the fines (-180 µm) from the glass sand production was insufficient for matching the fineness of the Ottawa sand. Therefore, some of the top size material of the Magdalen sand was removed using a 350 µm screen. To attain the desired AFS Number 52 while having an effective screening, two passes were necessary due to the static charge on the grains which caused them to adhere. The rejected coarse product, which contains 0.23% Al_2O_3, 0.05% K_2O and 0.09% Fe_2O_3, has visually little iron coating and might serve as glass sand after a magnetic separation. It has also a good potential as blast sand with an AFS Grain Fineness Number 40 prior to the 9420 psi test, and Number 62 after (as reported by Dieter Laboratory).

Comparative screen tests made on the unclassified and sized silica concentrates, as well as on a representative sample of Ottawa sand are reported in Table 34 with the corresponding AFS Grain Fineness Number.

Foundry tests

A 300-kg lot of classified foundry sand was submitted to the Foundry Section of CANMET for evaluation. From this lot a head sample was riffled for chemical analyses which are given in Table 35.

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The remainder of the head sample was submitted to Harry W. Dieter Co. to determine the physical constants of the foundry sand (21). The information is provided in Table 36.

The foundry group at CANMET prepared clay bonded sand moulds using both the Magdalen processed sand and Ottawa sand of the same grain fineness for repeated steel castings. The details of the work appears in a report by Szabo et al (22). Results are summarized in Table 37.

TABLE 33: Data for Mill Run II (foundry sand production)

I - Flotation conditions and reagents

(Feed rate: 70 kg/h, dry basis)

Operation	Time min	Dilution % solids	Reagents-kg/tonne H_2SO_4	PDD	PS	pH_i	pH_f
Rougher circuit:							
Conditioning	8	57 - 60	1.0	0.42		1.8	2.2-2.4
Flotation	1.8	30_i-24_f	0.7	-		2.2-2.4	2.3-2.4
Cleaner circuit:							
Conditioning	4	60	0.04	-		2.2-2.3	2.3-2.4
Flotation	0.7	10_i-6_f	0.42	-		2.3-2.4	2.4-2.5

II - Material balance

Products	Weight (%)	Analysis (%) Al_2O_3	Fe_2O_3	Quartz*	Distribution (%) Al_2O_3	Fe_2O_3	Quartz
SiO_2 conc	82.1	0.25	0.12	98.5	10.1	46.2	91.4
Al-Fe cl conc	10.7	15.35	0.60	16.6	81.2	30.1	2.0
Al-Fe cl tail	4.5	1.51	0.55	90.3	3.3	11.6	4.6
Coarse waste**	2.2	4.16	0.67	65.0	4.5	7.0	1.6
Fine waste	0.5	3.64	2.08	77.0	0.9	5.1	0.4
Feed	100.0	2.02	0.21	88.5	100.0	100.0	100.0

Symbols: $_i$ = initial and $_f$ = final

* Calculated

** Composed of 0.2% sea shell, 0.1% oversize sand and 1.9% misplaced grains.

TABLE 34: Screen analysis of foundry sands

Sieve		Magdalen Sand Weight (%)		Ottawa Sand Weight (%)
Metric µm	U.S. Standard Number	Unclassified	Sized	
590	30	0.0	0.0	0.2
420	40	0.3	0.3	3.3
297	50	37.8	21.5	30.1
210	70	45.8	57.4	42.2
149	100	15.4	20.2	22.4
105	140	0.4	0.5	1.7
74	200	0.1	0.1	0.1
53	270	0.0	0.0	0.0
-	pan	0.0	0.0	0.0
AFS Fineness Number		49	52	52

TABLE 35: Chemical analysis of foundry sand

SiO_2	Al_2O_3	K_2O	Na_2O	CaO	MgO	Fe_2O_3	TiO_2	LOI
99.3	0.26*	0.09	0.05	<0.1	<0.1	0.11	0.031	0.22

* Average of four analyses

TABLE 36: Physical characteristics of foundry sand

AFS Grain Fineness Number	51.5
Specific Surface (extrapolated)	111 cm^2/g
Coefficient of Angularity	1.15
"B" Sintering Point	1615°C
pH Determination	6.4
Durability at 9420 psi (measured by resulting AFS Number)	67.4*

* Under similar treatment the Ottawa sand increased to AFS Number 74.0.

TABLE 37: Selected properties of moulding mixes with Magdalen and Ottawa sands*

Moulding Data	Casting					
	No. 1		No. 3**		No. 5**	
	Magdalen	Ottawa	Magdalen	Ottawa	Magdalen	Ottawa
Sand (kg)	68.2	68.2	68.2	68.2	68.2	68.2
Clay (g)	4423	4423	56.7	255.2	170.1	170.1
Cereal	680.4	680.4	-	56.7	28.3	28.3
Moisture (%)	3.1	3.0	3.0	2.8	2.8	3.0
Mouldability Index	75	75	75	71	76	76
Permeability	191	214	221	240	222	236
Green Compression (kPa)	76.5	77.2	58.4	78.5	64.8	64.8
Green Shear (kPa)	26.9	22.7	22.0	24.8	22.7	21.4
Green Tensile (kPa)	11.5	13.2	12.2	11.7	10.6	10.6
Hot Tensile (kPa)	4.6	2.9	2.3	3.4	1.6	2.8
Dry Compression (kPa)	347	303	263	248	267	241
Mould Hardness	85-90	85-90	85-90	85-90	90-92	85-90
Methylene Consumption:						
i) before (mL)	28.2	31.0	29.8	31.6	28.3	30.6
ii) after (mL)	26.1	28.2	27.5	29.7	-	28.4

* From Dr. Szabo's report. The original figures were in Imperial units and have been converted to the metric system using 1 lb = 454 g, 1 psi = 6.89 kPa and 1 oz/si = 0.431 kPa.

** Re-cycling sand from previous castings.

These results show that the properties of the processed Magdalen sand are comparable to those of Ottawa sand. None of the results deteriorated significantly after four re-cyclings of the bonded sand. Behaviour during moulding and casting with the Magdalen sand was also comparable to the reference material, with the exception of the third casting in which moderate sintering occurred in the wedge area of the mould. Apart

from this exception, the other castings shook out easily and cleanly. Dr. Szabo attributed this to the iron coating on the grains. Subsequent testwork showed that this thin coating could be eliminated either partially by alkali scrubbing or totally by acid leaching. The CANMET foundry specialists concluded: "The (Magdalen) sand thus purified is likely to be able to meet the most rigorous demands that sand can be expected to encounter in the foundry" (22).

Feldspar Concentrate and Other Products

During the two mill runs some 75 kg of cleaner feldspar-iron concentrate was produced at an average grade of 15.2% Al_2O_3 which is sufficient for abrasive, extender and construction material applications. Part of it was kept for future testwork which will be concerned with attaining ceramic grade, i.e., 18.5% Al_2O_3, 11.5% K_2O, 2.5% Na_2O and less than 0.1% Fe_2O_3.

The feldspar-iron cleaner tailing analysed 2.0% Al_2O_3, 0.55% Fe_2O_3 and 85 to 90% quartz, which should be suitable for fiberglass manufacturing. Retreatment by flotation should produce a product of sufficient quality for either coloured glass sand or foundry sand. The treatment might also need a magnetic separation stage to reduce the high iron content. Since the quartz available in this product accounts for less than 5% of the total, such a study was not undertaken at this time.

DISCUSSION AND CONCLUSIONS

The main purpose of this work was to develop a technically feasible process for beneficiating raw marine sand economically and safely (from a health and environmental point of view) and secondly, to produce 300 kg of foundry sand and 200 kg of glassmaking silica from Magdalen Islands sand using the method developed.

It has been demonstrated that the collector N-tallow-1,3 propanediamine dioleate is a selective collector for feldspar. Futhermore with this collector it is not necessary to use the hazardous chemical hydofluoric acid which normally is used in feldspar-quartz flotation to depress quartz while floating feldspar. By controlling the pH within a

narrow range and using this selective feldspar collector, it was possible to produce a sand suitable for the foundry with undesirable alkalis reduced as shown below.

Product	Al_2O_3 %	K_2O %	Na_2O %	CaO %	Quartz %
Raw Sand	2.16	1.13	0.30	0.16	88
Foundry Sand	0.26	0.09	0.05	0.1	98

The sand was processed with minimal equipment, namely, three screens, one conditioner and limited flotation capacity (less than 2 min retention time). The reagent consumption was also low at 0.42 kg of collector and 2.2 kg of sulphuric acid per tonne of sand treated. This acid, in addition to being less dangerous than hydrofluoric acid, is considerably cheaper. Limited testwork also showed the possibility of partially recirculating the acidic water for additional savings. The metallurgy did not suffer because of these economies. Overall quartz recovery was 91% with less than 10% of the alkalis reporting in the quartz concentrate.

The glass sand was processed using a similar flowsheet and conditions except that a secondary collector was added, in small amounts (0.1 kg/t), to remove the metal oxides and a sulphuric acid leach stage was performed on the silica flotation product to remove iron oxide coating (about 0.1% Fe_2O_3). To minimize the cost of the sulphuric acid leach, multiple reuse of the acidic solution was investigated. More than 10 cycles of reutilization are possible for important savings in acid and neutralizing agent. In addition to feldspar rejection, the method used for processing the glass sand eliminated most of the metal oxides and other impurities, as evidenced hereunder:

Element	Raw sand	Glass sand
(%)		
SiO_2 (total)	95	99.6
Al_2O_3	2.16	0.2
K_2O	1.13	< 0.07
Na_2O	0.3	< 0.03
CaO	0.16	< 0.01
MgO	0.15	< 0.05
Fe_2O_3	0.23	0.025
TiO_2	0.05	0.02
LOI	0.3	0.1
C	0.04	0.02
ZrO_2	<0.02	< 0.01
(ppm)		
Chlorite	110	30
Mn	25	4
Ni	22	< 1
Cr	9	2
Cu	2	< 1
V	2	< 2
Co	1	< 1

These results are as good as could be expected when the mineralogy of the deposit is taken into consideration. Part of the feldspar and iron minerals are intergrown with and are present as inclusions in the quartz. Nevertheless, a further small improvement of the sand quality was still possible but required expensive hydrochloric acid leaching or high gradient magnetic separation. The utilization of these techniques would necessitate a high premium for the sand so treated. These two approaches were therefore not fully investigated.

Finally, some 80% of the feldspars contained in the raw sand were recovered at building material quality (15% Al_2O_3). Further testwork is scheduled to improve this concentrate to glass and ceramic grade quality. This will be described in a separate report.

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APPENDIX I

ANALYSIS OF SAMPLES FROM FLOTATION CIRCUIT OF MILL RUN I

APPENDIX I: Analysis of samples from flotation circuit of Mill Run I

Sampling		Analysis (%)					
		SiO_2 conc				Al-Fe ro conc	
Type	Period (h)	Al_2O_3	K_2O	Fe_2O_3	TiO_2	Al_2O_3	Fe_2O_3
Timed (1 min)	0 - ½	0.20	0.08	0.091	0.035	11.6	0.54
	½-1	0.21	0.08	0.094	0.032	10.8	0.49
	1 -1½	0.21	0.09	0.10	0.032	11.6	0.53
	1½-2	0.23	0.08	0.10	0.028	12.4	0.58
	2 -2½	0.21	0.06	0.10	0.026	13.1	0.60
	2½-3	0.22	0.07	0.10	0.027	12.6	0.60
	3 -3½	0.29	0.15	0.12	0.034	14.8	0.59
	3½-4	0.22	0.08	0.092	0.025	11.9	0.56
	4 -4½	0.22	0.08	0.10	0.027	11.6	0.56
Timed samples (averaged):		0.22	0.086	0.10	0.030	12.3	0.56
Bulk	0 -1½	0.20	0.07	0.11	0.029	-	-
	1½-3	0.22	0.07	0.10	0.028	-	-
	3 -4½	0.24	0.09	0.12	0.027	-	-
Bulk samples (averaged):		0.22	0.08	0.11	0.028	12.5	0.58
Overall average*:		0.22	0.083	0.105	0.029	12.4	0.57

* Used for material balance

Flotation feed analysis (%):

Al_2O_3	K_2O	CaO	MgO	Fe_2O_3	TiO_2
2.04	1.13	0.13	0.07	0.18	0.044

APPENDIX II

ANALYSIS OF SAMPLES FROM FLOTATION CIRCUIT OF MILL RUN II

APPENDIX II: Analysis of samples from flotation circuit of Mill Run II

Sampling		Analysis (%)					
		SiO_2 conc				Al-Fe ro conc	
Type	Period (h)	Al_2O_3	K_2O	Fe_2O_3	TiO_2	Al_2O_3	Fe_2O_3
Timed (1 min)	0 - ½	0.25	0.09	0.11	0.029	9.7	0.53
	½-1	0.24	0.09	0.11	0.034	11.6	0.51
	1 -1½	0.22	0.10	0.094	0.031	11.1	0.51
	1½-2	0.26	0.11	0.12	0.034	9.1	0.45
	2 -2½	0.30	0.14	0.14	0.035	11.6	0.49
	2½-3	0.28	0.12	0.11	0.028	11.4	0.55
	3 -3½	0.25	0.09	0.13	0.029	10.4	0.49
	3½-4	0.25	0.10	0.12	0.029	10.3	0.50
	4 -4½	0.25	0.11	0.11	0.029	10.4	0.49
	4½-5	0.25	0.09	0.12	0.030	11.1	0.56
	5 -5½	0.23	0.09	0.11	0.026	13.4	0.60
	5½-6	0.23	0.09	0.11	0.026	13.1	0.63
Timed samples (averaged):		0.25	0.10	0.12	0.030	11.2	0.53
Bulk	0 -1½	0.25	0.10	0.12	0.030	-	-
	1½-3	0.30	0.12	0.13	0.033	-	-
	3 -4½	0.26	0.12	0.12	0.030	-	-
	4½-6	0.23	0.08	0.11	0.030	-	-
Bulk samples (averaged):		0.26	0.10	0.12	0.031	12.4	0.53
Overall average*:		0.25	0.10	0.12	0.030	11.8	0.53

* Used for material balance

Flotation feed analysis (%):

Al_2O_3	K_2O	CaO	MgO	Fe_2O_3	TiO_2
2.03	1.11	0.14	0.07	0.18	0.040